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# AN IMPROVED EXPERIMENTAL UNIT FOR RECOVERY OF VOLATILE FLAVORS

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# AN IMPROVED EXPERIMENTAL UNIT FOR RECOVERY OF VOLATILE FLAVORS

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## BACKGROUND

In 1944 a process was developed at the Eastern Regional Research Laboratory for recovering the volatile flavors of apple juice in concentrated form known as "apple essence"(10)*. When this essence is added to concentrated apple juice a full-flavor concentrate is obtained (5, 7, 9). This flavor recovery process has since been applied to the preparation of high-density or frozen full-flavor fruit juice concentrates from a number of other fruits including grapes (4, 6), cherries (1), strawberries, peaches, raspberries, blueberries and blackberries. It has also been applied to the preparation of powdered grape juice (8) and powdered apple juice (13) and to the recovery of flavor from aroma-bearing vapors normally lost in preserve manufacture (2). Direct steam injection has been used as an alternative means for heating and vaporizing in the essence recovery process (3). The manufacture and use of volatile fruit flavor concentrates are subject to regulations of the U.S. Internal Revenue Service (14).

The essence recovery unit discussed in this publication is a modification of one described by G. W. M. Phillips et al, (12) with improvements based on subsequent studies. It is an experimental unit built with refinements for studying essence recovery from fruit juices and other aroma-containing liquids. It is equipped with facilities to permit tests under a wide variety of conditions in contrast to units designed for specific applications such as those referred to in references (2, 4, 6, 11). The unit is designed to minimize heat effect on the liquid being processed. Heating, vaporizing and cooling of the stripped liquid can be done at the equivalent of less than 3 seconds at 212° F. when the feed rate is 10 g. p. h. This is adequate to pasteurize fruit juices and to inactivate their enzymes. Arrangements are provided for prolonging the holding time where heat effects are desirable.

## GENERAL PRINCIPLES

When studies on the recovery of volatile flavors from fruit juices were extended to fruits other than apple, it became apparent that some modifications in equipment were required. There was designed therefore, a flexible experimental unit for essence recovery, with improvements which would overcome these difficulties as well as others that had been encountered, and at the same time would be capable of pasteurizing the juice without damaging its flavor by heat as conventional pasteurization often does. This unit was designed to have a capacity of 10 gallons per hour of juice when vaporizing up to 50% of the juice; or of 5.5 gallons per hour when vaporizing up to 90%. It should prove a valuable tool to facilitate varying the percentage of vaporization, the flow rate, the strength of the essence, and the heating time to determine the operating conditions best suited for any aroma-containing liquid.

The strength or volumetric "fold" of the essence is defined as the volume of juice processed in obtaining one volume of essence. If the stripping of the aroma from the juice and its recovery in the essence are both 100% efficient, the volumetric fold of the essence will represent the actual aroma potency with respect to the starting juice.

Figure 1 shows the arrangement diagrammatically. The fundamental principle of operation, as in the previous units developed at the Eastern Regional Research Laboratory, comprises rapidly vaporizing a portion of the juice to strip the aroma from it, fractionating

*Figures in parentheses refer to literature cited at end of the report.

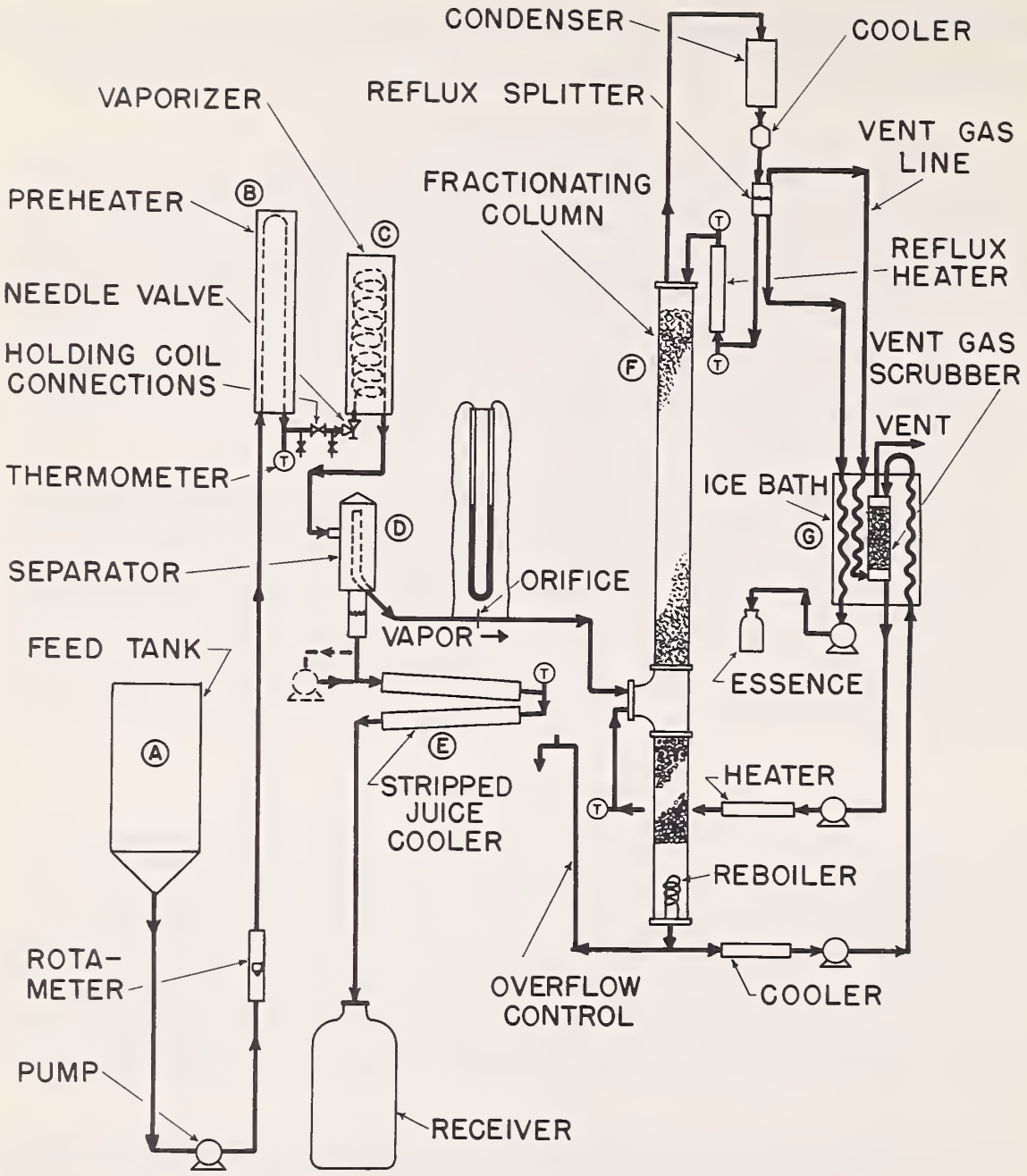


Figure 1. --Improved experimental unit for recovery of volatile flavors.

the vapor to concentrate the aroma into an aqueous essence, and scrubbing the vent gases from the condenser of the fractionation column with chilled liquid.

In this apparatus the juice is pumped from feed tank A (fig. 1) through preheater B and vaporizer C to separator D, where it is separated into stripped pasteurized juice and aroma-bearing vapor. The stripped juice is cooled in E and then collected for further processing. If too viscous for gravity flow, it is pumped through the cooler. The vapor from D passes to a fractionating column, F, equipped with the customary condenser, cooler, reflux splitter, and reflux reheater. A small portion of the reflux is pumped from the splitter, cooled, and collected as product ("essence"). The vent gases from the condenser are cooled, then scrubbed in G by countercurrent contact with chilled water. This water is obtained from the bottom of the fractionating column and, after being used for scrubbing, is returned to the column to recover the aroma.

## DETAILS OF APPARATUS

### Juice Feed

The liquid from which volatile flavor is to be recovered is charged through a conical fine-meshed screen into the stainless-steel feed tank, A, which is of about 12-gallon capacity. The screen should be of as fine a mesh as the liquid will pass through. The cover of the tank is tight fitting to minimize aroma loss. The liquid is delivered to the recovery system from the feed tank by a stainless-steel positive-delivery pump with adjustable speed, the rate of flow being metered through a rotameter calibrated for the particular juice used.

A positive-delivery pump is preferred over a centrifugal because the delivery of a positive pump is not seriously affected by accidental partial obstruction of the pipelines, and its uniformity of delivery helps to minimize surging in the single-tube evaporator. It is very important that juice be fed to the system at a constant rate, as the product (essence) is drawn off at a constant rate, and it is the ratio between feed rate and product-drawoff rate, after steady running conditions have been reached, that determines the strength (fold) of the essence.

### Evaporator

The evaporator consists of a preheater and a vaporizer. In earlier work juice heating and vaporization were done, for convenience, in a single tube, but later calculations predicted, and experience confirmed, that the time for heating and vaporizing and, hence, for the likelihood of heat damage is reduced if the juice is first separately heated in a tube of much smaller diameter than the vaporizer tube. Furthermore, uniformity of heating of all parts of the juice stream is achieved by choosing a preheater tube diameter small enough to ensure turbulent rather than streamline flow while the juice is in a harmful range of temperature. This avoids the long heating of the thin layer of juice which, under conditions of streamline flow, travels slowly along the wall of the tube. The inside diameter of the tube in the preheater is about as small as is practical, viz, 0.104 inch. Calculations show that applejuice of 14° Brix and 2.6 centipoises entering this preheater at 70° F. at a flow rate of 10 gallons per hour will be in turbulent flow, the Reynolds number being 2080. At a flow of 5 gallons per hour, the Reynolds number near the entrance is only 1040 and consequently streamline flow exists at that point; however, as the juice becomes heated its viscosity decreases and the Reynolds number increases until at the midpoint of the tube it is about 2400 and turbulent flow commences. Thus in the high-temperature portion of the preheater, local overheating is reduced.

The steam pressure on the jacket of preheater B is controlled so as to heat the juice to a temperature just above its boiling point at the pressure existing as it enters the vaporizer. The hot juice then enters vaporizer C, which has a 0.527-inch, inside diameter, tube. Sufficient steam pressure is used in the jacket of C to vaporize the desired percentage. For each kind of liquid a different percentage of vaporization is required to release substantially all the volatiles; for applejuice about 8% is adequate, for some fruits as

much as 50% is required, and for the distillates from the vacuum preserve kettle 30-40% is best. The vaporization in C is measured by metering the rate of flow of vapor from the liquid-vapor separator, D, by means of an orifice plate and a manometer.

Using separate tubes for the preheating and vaporizing operations in the manner described enables each to be designed closely for its own function, allowing a much smaller diameter for the preheater tube. By this means and by careful design of every part of the juice passages, the authors have been able to reduce the time that the juice remains at high temperature in the apparatus to less than 3 seconds at a feed rate of 10 g. p. h. This minimizes the heat treatment that the juice receives in passing through the apparatus, and consequently minimizes flavor damage. Many fruit juices develop a cooked taste and aroma on being held at 210° to 215° F. (99° to 102° C.) for 15 to 30 seconds.

Pulsation of the flow through the evaporator, which has given trouble in some essence units which do preheating and evaporating in the same tube, is eliminated by the use of a separate preheater controlled to heat the juice a little above its boiling point, and by having the feed pump of the positive displacement type rather than centrifugal. In a single tube which is fed with cold juice and which is required to heat it and vaporize a portion of it, there is always a tendency to pulsation unless the tube is fed by a pump of constant flow.

In the case of highly pectinaceous juices, such as apple, fouling of the inner wall of the preheater tube may occur. With larger essence-recovery units this fouling is eliminated if the minimum velocity is 20 feet per second. The preheater of the experimental unit would require a 0.058-inch, inside diameter, tube to obtain this high velocity but such a small tube is considered impractical because it would be prone to stoppage with extraneous material. However, if fouling occurs the tubes can be cleaned by steaming out and by occasional cleaning with a caustic solution.

The juice is heated in the preheater to a few degrees above its boiling point at the existing pressure. As the juice passes through the needle valve a relatively small percentage of the liquid flashes to vapor, insuring increased velocity and immediate boiling in the vaporizer and thus reducing the opportunity for heat damage or fouling at the start of the vaporizing tubes.

## Holding Coil

Because the flavor may actually be intensified by prolonged heating of certain fruit juices, for example, cherry juice (1), connections are provided between B and C for a jacketed holding coil which can be of any length required to achieve the desired holding time. This coil also enables the determination of maximum time permitted by heat-damage considerations; this figure is useful in design of commercial plants.

## Stripped Juice Cooler

The mixture of liquid and vapor from the vaporizer is separated in D, from the bottom of which the stripped juice flows to a two-stage cooler, E. The liquid level in the sightglass underneath separator D should be kept low to reduce the holdup time and thus minimize heat damage. Gravity flow through the cooler is used if the viscosity of the stripped juice is relatively low, such as that obtained with most juices when 50% or less is vaporized. However, a pump is required to force the stripped juice through the cooler if the viscosity is relatively high, as it is for most juices when more than 50% is vaporized. In order to cool the juice quickly to a harmless temperature, the tube in the first section of the cooler is of small diameter, 0.152 inch inside. The high velocity here increases the heat-transfer coefficient as well as decreases the contained volume, but causes a large friction drop. The latter part of the cooler, where temperatures are harmless, is of larger diameter, 0.402 inch inside. Cold water is fed through the jackets of the two coolers in series, parallel to the flow of the juice, to increase the rapidity of

the initial cooling. The cold stripped juice is received in a stoppered carboy for subsequent processing into a concentrate. These considerations do not, of course, apply to distillates and the like from which aroma has been stripped.

As an alternate method, the stripped juice could be cooled by flashing into an evacuated receiver; this would give practically instantaneous cooling. However, the tubular-type cooler is used in this unit so that studies can be made to determine the required amount of vaporization. This necessitates collecting samples of the stripped juice without the further loss of aroma that would occur with vacuum flash cooling. Flash cooling can be used in commercial units.

## Flavor Rectifier

The vapors leaving the liquid-vapor separator are already partially concentrated with respect to volatile-flavor constituents. For further concentration they enter the fractionating column, F, above the stripping section. This is a 4-inch, inside diameter, Pyrex glass pipe, the upper 52 inches of which (the enriching section) are packed with 1/4-inch ceramic Raschig rings. The lower part (the stripping section) contains 14 inches of the same packing. The maximum vapor capacity of the fractionating column is the vaporization of 5 gallons per hour of liquid. The feed rate and corresponding vaporization rate must be adjusted below this column capacity. In the previous experimental unit (12) the column diameter was 2 inches, but this required such low juice-feed rates (when vaporizing high percentages) that the juice might be heat damaged in the preheater. Below the stripping section an electric heater is immersed in a pool of liquid, forming a reboiler. In the previous experimental until (12) the stripping section was 6 inches long and the reboiler was 0.5 KW. Experience with that unit has shown there was a loss of aroma-bearing constituents in the liquid leaving the bottom of the column, when recovering the aromas of some fruits such as Concord grapes where the essence is not as readily volatilized as is apple essence. In order to decrease this loss the stripping section has been increased to 14 inches in the present unit and the reboiler increased to 2 KW. The increased number of theoretical stripping plates and the larger quantity of stripping steam should tend to force these constituents overhead into the essence.

Vapors leaving the top of the column are condensed, cooled to about 90° F. in the cooler, and passed to a glass reflux splitter; from this the desired quantity of condensate is drawn off as finished essence, and the remainder is reheated to about 190° F. and returned as reflux to the column. For example, in making 150-fold essence from apple-juice, if the juice feed rate is 10 gallons per hour, and the vaporization 10%, 1 gallon per hour would be vaporized and 0.067 gallon per hour would be drawn off as essence, leaving 0.933 gallon per hour to be returned as reflux. The "reflux ratio" would thus be approximately 14 to 1. The product (essence) is drawn off from the reflux splitter by a metering-type pump which controls the essence rate and pumps the essence through an ice-jacketed coil to a chilled receiver. This is preferable to the rotameter previously recommended. When the unit is started the concentration of volatiles in the overhead system is less than 150-fold. The column must be operated on total reflux long enough to reach the desired concentration.

## Vent Gas Scrubber

Because the starting liquid will contain a certain amount of dissolved air, which must be vented from the system and which will be saturated with volatile aromas at the temperature at which it leaves the reflux splitter, this air must be cooled and scrubbed before it is discarded. When commercially processing most fruit juices into essences of moderate (e. g. 150) fold, cooling and scrubbing of vent gases can be conveniently and effectively accomplished using the chilled essence itself as the scrubbing agent (6). In the arrangement shown here, the vapors from the column are condensed and cooled to about 90° F., the reflux being reheated before returning it to the column. The vent gases are scrubbed with chilled column bottoms instead of with essence. This procedure is highly effective and admirably suited for research purposes where short runs would preclude the accumulation of sufficient essence to use it as a scrubbing medium. On the other

hand, in large-scale operations the cooling and reheating of large volumes of condensate would be costly. The accumulation of essence to use as a scrubbing medium would constitute no serious problem in runs of long duration. If very high fold essence (e. g., above 350) is to be made on a commercial scale, cooling and reheating of the condensate and use of column-bottoms scrubbing may be necessary for efficient recovery of flavor top notes.

In Figure 1 the scrubbing column, G, is a 1-inch stainless-steel pipe, 12 inches long, packed with 1/4-inch Berl saddles. The chilled column bottoms enter the top of the scrubber, and the vent gases, previously chilled by passing through a coil immersed in the ice bath, enter the bottom and pass up in countercurrent contact with the liquid. Adequate scrubbing of the vent gases is obtained when the quantity of column bottoms used is sufficient to wet the packing thoroughly. The liquor from scrubbing column flows by gravity through a small heater, where it is heated to about 150° F. and then enters fractionating column F at the top of the stripping section, in order that the aroma which it contains may be concentrated and recovered as essence.

All parts of the equipment which come in contact with the juice, the vapors, or the essence are made of stainless steel, glass, neoprene, or, in the case of the packing, ceramics.

Detailed construction drawings for this unit may be obtained by writing to the Eastern Utilization Research and Development Division, Agricultural Research Service, U.S. Department of Agriculture, Philadelphia 18, Pa.

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